# Laboratory of Powder and Crystal Chemistry (Special Issue on the Commemoration of the Fiftieth Anniversary)

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# LABORATORY OF POWDER AND CRYSTAL CHEMISTRY

**Head: Dr. Natsu Uyeda**

The Laboratory established by Prof. E. Suito in 1952 has been succeeded to Prof. N. Uyeda in 1976 in order to develop the new field of science concerning the physical chemistry of powders and micro-crystals by the collaboration of staff members, Dr. M. Arakawa, Dr. T. Kobayashi, Dr. M. Nishino, and Mr. T. Yoshida. The scopes of major researches are outlined hereunder together with the lists of papers published during the last decade.

## I. Transformation and Growth of Microcrystals in Suspensions

It was found by X-ray diffraction and electron microscopy that metastable powders of organic compounds are transformed to stable forms, being accompanied by considerable crystal growth in organic suspensions. This phenomenon is common to various metal derivatives of phthalocyanine. Remarkable solvent effects were also observed giving rise to intermediate states during the transformation, including preliminary growth, formations of complexes with solvent molecules and different polymorphs. These effects were interpreted in terms of electron affinities and donating properties of dispersed powders and suspension media.

The crystal structures of some adducts complexes of Zn and Fe-phthalocyanines were determined by X-ray diffraction analysis.

## II. Epitaxial Growth and Topotactic Formation of Thin Films of Organic Semi-Conductors and Charge Transfer Complexes

Electron microscopy was applied to the studies of thin crystalline films formed on cleaved surfaces of ionic crystals by epitaxial growth from the vapor phase. The orientation and structures were analysed by the combined use of selected area diffraction to find that there are certain evidences for the crystal orientation being mostly controlled by the oriented adsorption of molecules which form the epitactic nucleation on the substrate surfaces.

LEED and AES were also applied to confirm the state of oriented adsorption with systematic combinations of alkali halides as substrates and cyano- and halogeno-derivatives of ethylene as absorbates.

Topotactic formation of charge transfer complexes was also studied with TCNQ as electron acceptor and counterpart donnors such as metal ions and anthracene.

## III. High Resolution Electron Microscopy of Fine Structures of Microcrystals

Various lattice defects, such as multiple twins, spiral steps, dislocations, and stacking faults were investigated by high resolution electron microscopy with respect to colloidal particles of gold and silver, and various clay minerals. The fine structures were analysed by the observation of diffraction contrasts, moiré fringes and lattice images which directly reveal the arrangements in crystals.

## IV. Direct Observation of Molecular Images for the Structure Determination

Since the first success in the direct observation of molecular images of hexadecachloro-Cu-Phthalocyanine arrayed in thin epitaxial films, high resolution electron microscopy of atomic order has been explored from both experimental and theoretical phases. The qualification of molecular images and the reconstruction of correct structures have been undertaken by means of computer simulation.

Joint exploration on the attainment of atomic resolution with Prof. K. Kobayashi, is also proceeded in regard to the construction and application of new 500 kV electron microscope.

## V. Characteristics of Powder

Systematic investigations have been carried out concerning the physical properties of various industrial powders in view of the characteristic behavior unexpectable from bulk properties. The mechanical properties such as fluidity, packing property and mixing of powders have been investigated in relation to the shape, size, surface state, and interaction of component particles. The methods to estimate these properties are also explored with model powders, practical application to the analysis, and the classification of results.

## Publications

(* indicates an article published in Japanese)

### I. Transformation and Growth of Microcrystals in Suspensions


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## II. Epitaxial Growth and Topotactic Formation of Thin Films of Organic Semi-Conductors and Charge Transfer Complexes

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3. T. Takenaka, S. Tadokoro, and N. Uyeda: Infrared Absorption Spectra of Tetracyanoethylene Adsorbed on Evaporated Alkali Halides., *Bull. Inst. Chem. Res., Kyoto Univ.*, **48**, 249 (1970).
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